Notebook Series -- Notebooks by Experimenters Other Than Edison
Chemical Experiments -- L. F. Ott Books
Notebook, N-14-05-19

This notebook was used by Ludwig F. (Louis) Ott during May 1914-February 1915 for notes on various chemical experiments. Included are experiments on paraphenylenediamine and Condensite for disc records and on nickel flake and nickel oxide for storage batteries. Other experiments relate to condensation experiments undertaken for Edison, the recovery of nickel flake and nickel oxide from old battery cells at the Edison Storage Battery Co., and the use of Condensite to replace celluloid on Blue Amberol records. In addition, there are notes pertaining to fireproof fabric for the kinetophone studio, dust-laying compounds for cement floors, and specifications for toluol from the British government. A few of the entries bear notations by Edison. The last page is an index to the experiments in the book. The index has been filmed first. The front cover is labeled "Note Book." The book contains 160 numbered pages, some of which are blank.

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60912

Irome Co.,

MFG. STATIONERS,
96 JOHN ST.
AND
19 PLATT ST.
NEW YORK.

Note Book of
Ludwig Ott
From May 19, 1914.

May 19, 1914.

Experiment on Recovery of Nickel Flake and Nickel Oxide from Old Cells from Edison Storage Battery Co.

1

In all cases ~~grams of~~ Solutions were used if the Edison was put in a solution of 160 CCm of Water + 40 grams of the following salts unless otherwise specified Approximately a 20% Solution. Given to Mr. Edison to test the Ni Plate

400 K. Carbonate ✓

401 K. Oxalate ✓

402 K. Perchlorate ← over half undissolved

403 K. Arsenite ✓

404 K. Benzoate ✓

405 K. Chlorate ← about 1/2 soluble

406 K. Sulphite ← about 3/4 Soluble

407 K. Nitrite ✓

408 K. Chloride ✓

409 K. Sulphate ← about 3/4 soluble

410 K. Bisulphate ✓

411 K Ferrocyanide ✓

412 K. Hypophosphorus

413 K. Ferricyanide ✓

414 K. Lactate

415 K. Ethyl Sulphate

416 K. Persulphate

417 K. Ethylsulphonate

418 K. Carbolate ✓

419 K. Formate ✓

420 K. Citrate ✓

421 K. Lactophosphate 30 grams
Looks questionable

422 K. Sulph ethylate

423 K. Meta Borate

424 K. Sulphocyanate ✓

425 K. Bromide ✓

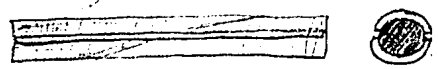
5

426 K Benzenedisulphonate 16 grams

427 K. Sulphobenzoate

May 21, 14 6

Experiment to see if the following solutions which discoloured the metal from Old Tubes will [illegible] two halves of a tube to separate when they are sawed fast way on [illegible] tubes



428 Sodium Hypophosphite Bright Tube
429 " " Yellow Tube
Both in Thursday May 21 at 12.00 noon

430 Hydrogen Peroxide in Thursday May 21 at 1200 from Reward Sol Blumer,

431 K. Bichromate in May 22 430 P.M.

432 K. Iodide May 22 4.30 P.M.

433 K. Ferrocyanide May 22 4.30 P.M.

434 K. Sulphide (Liver) May 22 4.30 P.M.

435 K. Hypophosphorus May 22 4.30 P.M.

436 Na Sulphite May 22 4.30 P.M.

437 Na Sulphate May 22 4.30 P.M.

438 Na Salicylate May 22 4.30 P.M.

May 22/14 #439
Put on 4 Tubes 1 Bright
1 extra brown the rest not selected
in Hot water (Hot Plate) at 5.00 P.M.
to see if the tubes will separate from the Milk.
Left on Hot Plat until May 28, 5.00 P.M.

440 Potassium Oxalate

441 K Arsenite

442 K Bitartrate

443 K. Sulphocyanate

444 Na Nitrite

445 Na Chloride in May 25 1.00 PM

446 K. Chlorate in May 25 1.00 PM

447 Na & K Sulphate left out on account of the Al

448 3 tubes 1 brown Put in 5% Glycerin with a few drops (about 20) 20% Na OH on May. 25, at 10.30 AM Hot Plate off May 29 at 4.00 PM.

#449 6 Tubes in 25% Na Hypo-sulphite May 26, 10 AM

450 6 Tubes in 25% Sol Sodium Sulphite. May 26, 10 AM

451 Same solution as #449 except added 10 drops 20% NaOH on May 6, 1.30 PM

From 452 to 468 incl. May 26 at 4 AM

452 K. Carbonate Both tubes open just a little Sol clear no color Half dried out June 2 10 AM

453 K. Arsenite Both tubes open 1/16 inch sol clear no color Nearly dried out June 2 10 AM

454 K. Nitrite Tubes open but very little sol clear no color Half dried out June 2 10 AM.

455 K. Chloride Pockets do not open much just deal of juice, both ferrous and ferric? Half dried out June 2 10.0 AM

456

457 K. Permanganate Pockets open but very little No good

458 K. Persulphate Tubes open a little Tubes twisted around with green crystals sol. green.

459 K. Hyposulphite Tubes ready to fall apart. Promising
some black sediment also some Ferric Iron. not as much Ferric as the Na salt. Duplicate with 4 Tubes on May 29 3.30 P.M.

460 K. Ferricyanide N. G.
No good oxidized

461 K. Salicylate Tubes open a little
Half does it out June 2 10 a.m.

462 K Sulphocyanide N. G some Ferric
Too much iron.

463 Sequette Salts Tubes open about 1/16 inch
some green flocculent Precip. Not good

464 Na Phenylate Tubes open about 1/16 inch
3/4 disintegrated promising.

465 Na Borate ~~Na~~ Not Good

~~466 Na Oxalate [illegible]~~

467 Na. Peroxide N. G.
1/2 disintegrated Clean.

468 Na Bichromate Tubes open but very little
N. G.

469 K. Formate on May 28 2.30 P.M
Tubes open up about 1/16" sol slightly green. clean
very promising.

470 K Sulphite on May 28 2.30 P.M
Sol very yellow (muddy) not promising

471 5% Sol of Sulphurous Acid sol
very green Fair / oxidizes out June 2 10 a.m.

472 K. Citrate May 29. 12.15 - p.m.
Nearly does it out June 2 10 a.m.

473 Na Sulphide Nitro May 29. 12.15 - p.m.
Nearly does it out June 2. 10 a.m.

474 Na Nitrite May. 29. 12.15. p.m.
No good

475 Na Manganate May 29 - 12 - 15 - p.m.
No good

476 Na Lactate May 29 - 12 - 15 p.m.
3/4 disintegrated clean. / tubes easily separated
Promising.

477 K Carbolate May 29 - 12 - 15 p.m.
3/4 disintegrated clean promising

478 Na & K. Sulphate May 29 - 12 - 15 p.m.
Too much action no good

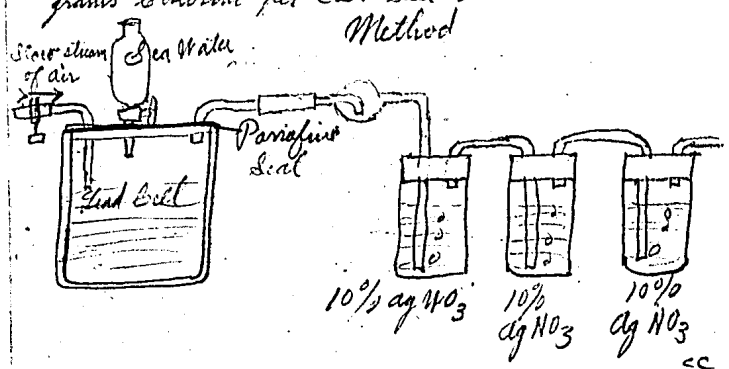
479 Iron from old packets 200 grams
in 175 cc 30% Sulphuric Acid on
4 days June 5, 14.
Duplicate on. 5.00 PM June 8, 14.

June 15/14

Experiment on the evolving of Chlorine Gas when Sea Water enters a Lead Cell also in Edison Cell

Sea Water 50 cc Chlorine 34.5 cc 10% $AgNO_3$
Precipitating with $AgNO_3$ gave grams
$AgCl$ corresponding to grams of Chlorine
as chlorides in 50 cc Sea water or
grams Chlorine per cc. Sea Water.

Method



Lead Cell 3/4 Full of Solution let in 100 cc Sea Water the Gases evolved were passed through 3 batteries of jars containing 10% Solution of $AgNO_3$ then passed air through to drive out the Chlorine remaining in the cell. The Solution in the 3 batteries of $AgNO_3$ was then filtered off and weighed on a dried and weighed filter.

100 cc Sea Water in the cell passed through 1st battery containing cc 10% $AgNO_3$ enough to absorb the total amount of Chlorine evolved 2nd and 3rd batteries contained cc 10% $AgNO_3$ total cc. 10% $AgNO_3$

Weight Total = grams
" Filter = _____ grams
" $AgCl$ = grams

$AgCl$ = 143.4 Cl = 35.5

X : 35.5 :: 2.8528 : 143.4

Grams $AgCl$ Found 2.8528

35.5
142640
142640
85584
143.4.) 1012.7440 (.706 grams chlorine
10038
8944
in the Sea Water

or 1.512 grams per 100 cc

152420
14.3510
.8910

Sea Water alone

18.0948
14.3510
3.7438

Total = 37438
Filter = .8910
$AgCl$ = 2.8528

Analysis of WYOIL

2 sample bottles received July 7, 14 from Mr. Hutchinson

Sp. Gravity @ 20°C = 0.8625

Viscosity at 70°F 1st time 1 min 8 4/5 sec
" " " 2nd " 1 " 8 3/5 sec
" " " 3rd " 1 " 8 2/5 sec
Average of Viscosities at 70°F = 1 min 8 3/5 sec or 68 3/5 sec

Viscosity at 212°F. 1st time 19 sec
" " " " 2nd " 19 sec
" " " " 3rd " 19 sec
Average of viscosities at 210°F = 19 sec.

Flash Point = 310°F

Saponification Figure none

39.5269 Total
37.6810 Flask
1.8759 g Oil + 27 cc n/2 Alcoholic KOH boiled 1 hour

gave 55.6
30.
27 cc KOH require 25.6 cc n/2 H_2SO_4

Blank Test 81.6
56.0
27 cc KOH required 25.6 cc n/2 H_2SO_4

Acidity —
5 cc Oil require 0.1 cc n/2 Alcoholic KOH =
= 0.00245 H_2SO_4
100 cc = 0.049 g H_2SO_4 or practically
5/100 of 1% Free Acid.

Iodine Figure

Total 45.3090
Blank 44.4355
Oil .8735

Iodine solution = 25 g Iodine, 30 g $HgCl_2$ dissolved separately to 500 cc with 95% Alcohol. Mixed & stood 2 days then diluted the oil in 10 cc Chloroform added 25 cc Iodine solution let stand 6 hours then added 20 cc 10% KI Sol. 300 cc H_2O and titrated with n/2 Thiosulphate Sol. [illegible]

Blank required 68.2 cc n/2 Thiosulphate Sol
Oil " 68.25 cc n/2 " "

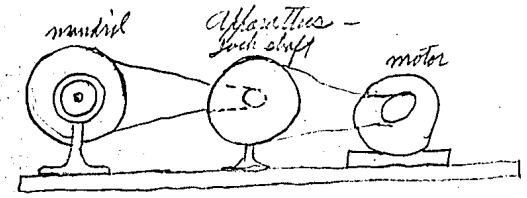
after heating 10 hours at 212° F. and passing a good stream of air through the oil. Viscosity is as follows

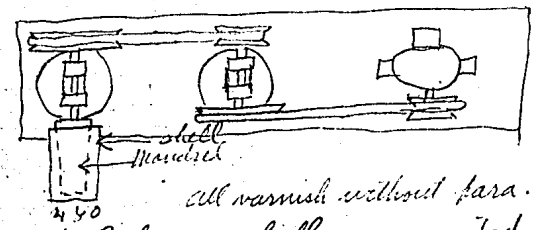
1st	1 min 9 sec.
2nd	1 min 8 $\frac{4}{5}$ sec
3rd	1 min 8 $\frac{4}{5}$ sec
Say	1 min 8 $\frac{4}{5}$ sec Average

this is $\frac{1}{5}$ sec. greater viscosity than it was before treatment.

July 16, 14

Experiment on a method of using condensite to replace the celluloid in Blue Ambrol Records Charge 2914.





All varnish without fara.

1st A brass shell was coated with condensite varnish by dipping, then put on the mandrel and dried by rotating at 30 rev per Min. This would not work as the varnish had a tendency to shrink in rings towards the end which the airplings came off.

-481

Then tried to brush the varnish on the shell while in rotation this worked very good but in baking the varnish sticks to the shell so hard that it would not

come off cold as by heating

482 Dried the same as above except first applied a trace of Sesame Oil with a rag on the shell before coating with varnish.

483 Wrapped 1 layer of thin waxed paper stuck together with varnish, dried. then coated 4 coats with the brush and fired varnish ran through paper in two spots so I had to slit it in order to remove from shell.

484 Brass shell dipped in paraffine then coated 4 coats with varnish by brushing on 1/2 hour between each coat with a final dry of 1 hour then fired according to schedule #1

Schedule #1

1 hour	to	100	
1 "	"	130	N.G. Gas bubbles
1 "	"	160	Paraffin melts at
1 "	"	190	
1 "	"	220	140 F.
hold 1 "	at	220	Temp. lowest = 160

485 Same as #484 ~~except~~ dipped first 1 end dipped ~~[illegible]~~ then dipped the other end cut. M.P. of Paraffine 140°F

486 Two layers manilla wrapping paper glued together with varnish taken off shell then dipped in varnish 1 part alcohol 1 part. and fired at 160°F all night. looks good no shrinkage. then coated 4 thickness of varnish not diluted then fired again as above coated with brush (bubbles crack too easily)

487 Tracing paper 7 Carrie Tracing Crane & Co. dipped in varnish 1 part alcohol 1 part and put through roller, description page 23. then fired all night 160°F. then wrapped around shell and glued with varnish then coated four coats varnish not dilute with brush baked 160°F all night (Bubbles crack easily)

488 ~~Shell~~ Shell dipped in a sol. of 20 grams Phenol resin 100 cc alcohol dried dipped again dried then coated 4 coats varnish with brush fired all night 160°F.

over

then the Phenol resin dissolved out in alcohol – became soft and all blisters –

489. Two layers of Flexine Tracing paper dipped in Sesame Oil Scraped, then joined with varnish. coated 4 coats varnish fired 160°F. all night. 1= after scraping wiped dry with towel – Bubbles cracks too easily

490 Shell dipped in regular celluloid dipping solution two dips dried, coated 4 coats varnish, brush, fired 160°F. all night. N.G. All blisters and cracks off. —

491 Shell dipped in Hot Phenol Resin hot to hot shell or the least change of temperature warps the resin fine coating bell cracks off on cooling probably due to contraction.

492 Duplicate of 488 except left in alcohol only a short time

493 Shell dipped in Zelva chlor naphthalene this does not adhere well to the brass seems to have some bare spots which will not cover: had to touch these up. ~~[illegible]~~ see 498

494 Shell coated with a solution of 20g Prime Cuban Asphalt in 100cc Benzole this coat fine. dipped twice first 1 end then the other.

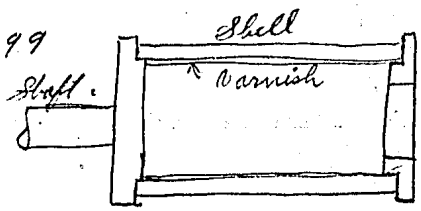
#495 Shell dipped in 20g Gilsonite dissolved in 100cc Benzole – dipped twice first 1 end then the other.

496 Same as # 494 except. 40 grams Prime Cuban Asphalt in 100cc Benzole

497 Same as # 495 except. 40 grams Gilsonite in 100cc Benzole.

498 Cold shell dipped in melted Zelva chlor naphthalene let stand about 1 min. on first dip, then cooled 5 min then dipped quickly 3 or 4 dips. then turned down smooth on lathe. then coated 6 to 8 coats varnish (regular) with brush on revolving mandrel baked 4 hrs at 100–120 then 1 hr to 140 then 1 hour to 160 all night 160 —
then the Zelva dissolved off with benzine or benzole.

499



the shell while rotating filled with 75cc Reg Varnish dried in air 4 hrs then gradually raised temp to 190°F. this gave a thick round blank but all bucketed. had to force out with a ring in the hydraulic press.
all inside parts coated with trace of Sesame Oil before varnishing

500 same as 499 except 25cc too thin to force out of brass shell, but smooth.

501 same as 499 thinner than 499 but still bucketed.

502 same as 500 except after finishing put in a core backed up with 1 to 1 powder mix pressed down firmly hard and baked at 320°F for 1/2 hour

503 same as 502 except used a richer mix than 1-to1-

29

On 10 am Monday

Condensation Exp for Mr. Edison

#1 30g Phenol.
504 12g 40% Formaldehyde

#2 30g Phenol.
505 12g 40% Formaldehyde
100 milligrams 6/4

#3 30g Phenol
506 12g 40% Formaldehyde
2 cc Dry Ammonia

30

#4 30g. Phenol
507 15g 40% Formaldehyde

#5 30g Phenol
508 15g. 40% Formaldehyde
115 milligrams 6/4
Went to Resin 5 pm. Wednesday

#6 30g Phenol
509 15g. 40% Formaldehyde
2 cc Dry Ammonia.
Went to resin Wednesday 10 am

#7 30g Phenol
510 20g 40% Formaldehyde

#8 30g Phenol
514 20g 40% Formaldehyde
100 mili grams 6/4
Went to ~~Retort~~ Resin ~~Thursday~~ 6 pm 8 hours

#9 30g Phenol
512 20g 40% Formaldehyde
2 cc. Reg. Ammonia

#10 30 g Mela Para Cresol
513 12 g 40% Formaldehyde
Resin at 6 pm 8 hours

#11 30 g Mela Para Cresol
514 12 g 40% Formaldehyde
100 mili grams 6/7
went to Resin - Tuesday am -

#12 30 g Mela Para Cresol
515 12 g 40% Formaldehyde
2 cc Reg Ammonia

#13 30g Mela Para Cresol
516 15g 40% Formaldehyde
went to Resin Tuesday am

#14 30g Mela Para Cresol
517 15g 40% Formaldehyde
10 c.c. nitric acid 6/4 — 5.5

#15 30 grams Mela Para Cresol
518 15g 40% Formaldehyde
2 c.c. Reg Ammonia

16 30g Mela Para Cresol
519 20g 40% Formaldehyde
Rubber at 6 pm — 8 hours

17 30g Mela Para Cresol
520 20g 40% Formaldehyde
100 [illegible]

18 30g Mela Para Cresol
521 20g 40% Formaldehyde
2 c.c. Reg. Ammonia

All ingredients obtained from Hoffman
Phenol is the Straight English Phenol
Cresol is the Bayway Mela Para
Hex is about [illegible] Hexamine
Formaldehyde 40%.
Ammonia CP stuff Sp. G. 0.9 = 28.3%
On hot plate Sept. 12, 14. at 11.30 P.M.
Temp. of hot plate = 240°F = 115°C

~~#20~~ Made saturated Solutions of the following.

K_2SO_4 K.Br. K_2CO_3 $KClO_3$
KCl. KNO_3

Mr. Edison ~~filled~~ put some Ni Hydrate #731 Lot Number into tumblers and treated it with the above sat. solutions all six put on shelf with covers on Sept. 17, 14, at 10.00 A.M.
Lot Number solution the Hydrate was treated with, together with date on Label on tumbler.

turned steam off 6 pm Sept 17

~~#20~~

#19 Treatment same as # 1 to 18 inclusive but 30 cc Phenol.
522 18 cc Formaldehyde 40%
Passed SO_2 for 5 minutes
then put on hot plate Sept. 17, 14. at 3.00 P.M. SO_2 made sol. with all reactions looks as if Sulphur Acid [illegible] – at 540 PM 2 hours later looks as of Resin – I cool it over night –

#20 Same treatment as 19 except.
523 30 g. Para
20 cc Formaldehyde 40%
on hot plate Sept. 17. 14, at 3.00 P.M.
no SO_2 – looks as of Condensed Some

524
#21 Same treatment as #19 except. 30 g Penta
20 cc Formaldehyde 40%
Put on hot plate Sept. 17, 14. at 3.00 P.M.
no SO_2

525
#22 100 g Straight English Phenol
5 g Dried 6/4 from Hoffman
on. Sept. 30 at 10.~~30~~ 15 a. m.

526
#23 100 g Straight English Phenol
10 g Dried 6/4 from Hoffman
on Sept. 30 at 10.~~30~~ 15 a. m.

527
#24 100 g Straight English Phenol
15 g Dried 6/4 from Hoffman
on. Sept. 30 at 10.~~30~~ 15 a. m.

528
#25 100 g Straight English Phenol
20 g Dried 6/4 from Hoffman.
on. Sept. 30 at 10.~~30~~ 15 a. m.

all put into ⌂ on hot plate.

#529 Sept. 29, 14.

Viscosity of varnish

Regular #903 Dob [illegible]

1st trial	4 min 28 sec	80° F
2nd trial	4 min 30 sec	" "
3rd trial	4 min 32	" "
Average	4 min 30 sec. at 80° F.	

#530
Regular varnish same as #529 [illegible]
700 grams varnish + 1 gram Acetone

1st trial	80° F
2nd trial	" "
3rd trial	" "
Average	" "

#531 Same varnish as #529 except
100 grams Varnish + 2 grams Acetone

1st trial
2nd "
3rd "
Average

#532 Same varnish as #529 except
100 grams varnish + 3 grams Acetone.

1st trial
2nd "
3rd "
Average.

#533 Same varnish as #529
except 100 grams varnish 4 grams Acetone

1st trial
2nd "
3rd "
Average

#534 Same varnish as #529 except,
100 grams varnish 5 grams Acetone

1st trial	2 min 50 sec.
2nd trial	2 min 56 sec
3rd trial	2 min 43 sec.
Average	2 min 43 sec

#535 Same as 529 except,
100 g varnish + 5 grams Alcohol (Methanol)

1st Trial	3 min 10 sec.	Temp 80°F.
2nd "	3 min 10 sec.	
3rd "	3 min. 10 sec.	

536 Same as 529 Trial of Regular varnish Sept. 30, 14.

1st	4 min	10 sec	Temp 80°F
2nd	4 "	10 "	" "
3rd	4 "	6 "	" "
Average	4 min	8 sec.	" "

#537 Same as # 529 except,
100 grams Varnish + 5 g Benzol

1st Trial	2 min. 52 sec	Temp. 80°F.
2nd Trial	2 min 50 sec.	" " "
3rd Trial	2 min 51 sec	" " "
average	2 min 51 sec.	" " "

#538 Same as # 529 except
100 grams Varnish + 5 grams Amyl Acetate Conc.

1st	3 min	5 sec
2nd	3 min	5 sec.
3rd	3 min	5 sec

Comparison of Viscosities taken at 80°F.

No.	Description	Special Varnish	*	Difference
534	100 g Varnish (Regular) + 5 g Acetone	2 min 43	4 min 30	1 min 47
535	100 g " " + 5 g Alcohol (Methanol)	3 min 10	4 min 30	1 min 20
537	100 g " " + 5 g Benzole	2 min 51	4 min 8	1 min 17
538	100 g " " + 5 g Acetic Acid Conc.	3 min 5	4 min 8	1 min 3

Experiment # 539

500 grams Aniline + 54 grams $NaNO_2$ + 100 cc H_2O were put in a lead vessel provided with a stirrer and surrounded with a water jacket, under stirring was added 350 cc 22 °Be HCl which took 5 hours temp. kept at 30–40 °C then raised temp. to 70–80 °C for 2 hours and put in 100 grams. Zinc. let go all night boiled up for 2 hours next morning, then filtered washing the solid part well with boiling water. then added 180–190 grams NaOH Primary Battly stuff. until slightly alkaline Filtered, evaporated to nearly dryness and dissolved in Alcohol this dissolved the para. leaving the formed Na. Cl. behind.

Exp. # 540

Same as 539 except used iron this forms basic salts. which oxadize the para too rapidly and are extremely hard to filter off when precip. with an alkali.

Exp. # 541

took Para nitro aniline reduced it with Tin and HCl precip the Tin with H_2S and filtered off boiled off H_2S, evaporated to cryst. gave the acid salt. ~~this should~~ Acid salt ~~fro~~ distilled with excess of caustic soda. giving the neutral basic para salt. which looks O.K. Glass melts eaten through in the distillation.

Exp. # 542

Same as above except. got rid of the Chloride acid salt by neutralizing with NaOH evaporating and dissolving the para in alcohol

Details of 542 and 543 on pages 47–48

#543
Same as #542 except used Zinc to reduce.

Details of 542
10g paranitroaniline dissolved in 55cc 22°Be HCl + 200cc H_2O then added 20 grams Tin

Note— The ~~para~~ reduction of the p-nitro aniline goes smoothly the precip of the reducing agents Tin or Zinc with NaOH and filtering goes smoothly, but not so with iron as Ferrous salts are formed ~~in the~~ which oxidize and at the same time oxidize the para and make it black.
The filtered liquor from the prec. of Tin or Zinc with NaOH is nearly white but rapidly gets dis-coloured

This liquor then contains ~~p-amido~~ p Phenylenediamine + NaCl: it is then evaporated to near dryness and the para dissolved out in alcohol leaving the NaCl behind.

544 -- 450 grams Hexamethylenetetra amine. was dissolved in 5000 cc hot alcohol from Hoffman then 250 to 300 cc conc. C.P. Hydrochloric Acid gradually added under constant stirring until no more precip. was formed in the clear alcoholic sol. by the addition of more HCl. when this point was reached the sol became acid to litmus before it was slightly alkaline. Filtered off alcohol. ~~which~~. alcohol ~~state~~ allowed to stand over night which gave on cooling the crystals marked 544 a that which remain in the retort after distilling off the alcohol is 544 b and the alcohol 544 c

545 Distillation of Penta extracted with Sodium Carbonate ~~and~~ by Dr Hye

at 100 °C some white crystals come over with the water.

130–135 very little brown crystalline stuff

140–150 very little crystalline stuff

200–210 very little crystalline stuff

280 Quite a little white crystally distillate

300 Some solid crystalline distillate (yellow)

Over 300 a white sublimate together with a yellow crystalline substance left in the neck of the retort, while a black residue remained strong smell of HCl.

All up to and including 280 soluble in alcohol. those above 280 not soluble in alcohol.

#546 ~~Sept~~ Oct. 3, 14

Distilled some Straight English Phenol from Hoffman 100 grams
No water Phenol came over at 178–180°C constantly ~~and~~ until the very last about 3 to 4 cc came over at 150–160 Dropped because there was not enough vapor to heat the thermometer the residue black like charcoal. not much residue. nearly 1/4 gram

#547 Oct. 5, 14.

Distilled Phenol from Silver Lake Batch #2 at 100°C 30 cc Phenol water came over which on settling gave 27 cc Water with 3 cc phenol on the bottom.

on the rise from 110° to 170° 2 cc came over (Phenol)

180–183°C	26 cc	phenol
185–189°C	13 cc	Phenol
190–195°C	8 cc	Phenol / probably ortho cresol)

195–200°C 3 cc probably meta + para Cresol.

Over 200 3 cc tarry oil

Therefore in 100 grams there are 100 grams = cc

Water	27 ~~X~~ cc
From 180–190°C Total amount reckoned as Phenol	44 cc
190 to over 200	11 cc
	.82 cc
Residue	18 g Residue

None of these fractions crystallize even on cooling.

#548 ~~Start~~ 100 rose gradually to 170 Phenol distilled over at a nearly constant temperature 178 – 180 About 2 cc phenol water not phenol only 1/2 cc coming over above 180 - 180-185. very little residue.

Exp. # 549 The Fractions from Exp. #547 Distilling over at 180-183 and 185-~~190~~ 189 respectively were mixed together and redistilled.

100 to 170	2 grams
175	4.5 grams
178 – 183	19. grams crystallizes (cooling on ice)
185 – 189	5 grams
190 and over	1 gram
Residue of about	1 to 2 grams left in retort

2335 / 310 (
31) 2335 / 217.0 / 18 (75%

550 100 cc of Phenol from Silver Lake was put into a graduate and 20 grams solid NaCl added last night water level stood at 26 cc This morning at 29 cc

Found 80 cc Phenol left after separating with separating funnel
Repeated whole operation.

551 25 cc Reg Varnish # 903/588 lot
100 mg Hard Phosphoric Acid

552 25 cc Reg Varnish # 903/588 lot
250 mg

553 25 cc Reg Varnish # 903/588 lot
500 mg

554 25 cc Reg Varnish # 903/588 lot
1 gram

555 25 cc Reg Varnish # 903/588 lot
2 grams

Exp. # 556 100 cc Phenol Lot # 2 from Silver Lake was just neutralized with a 20% Sol. of Na OH then enough 20% sol. of Na OH added to make about 5% Na OH in excess. This sol. was then diluted to 1000 cc with water. and allowed to settle. There remained undissolved 18 to 20 cc of an insoluble oily residue The dissolved portion was then poured off and made acid (slightly) with HCl Added 300 grams Na Cl and shook vigorously then let settle, drained off the water and found 50 cc Phenol

50 cc Phenol
20 cc oily Residue
30 cc Water from previous ~~tests~~ distillations
100 cc

Exp # 557 100 cc Phenol
Lot # 2 was mixed under constant agitation (rotary stirrer) with 400 cc Benzole for 1/2 hour then put into a seperatory funnel.
39 cc ~~Have~~ Remained insoluble
The benzole was then distilled off.
came over milky at first then cleared.

100° to 170° C	=	[illegible] cc	12 cc
183° to 184° C	=	[illegible] cc	28 cc
185° to 190° C	=	[illegible] cc	8 cc
190° to 200° C	=	[illegible] cc	4 cc
200° to 240° C	=	4 cc	4 cc
Residue over 240° C			5 cc

Exp. # 558 Distillation of Sample of Recovered Phenol from Hoffman
Weight of flask = 55.5 g.
Weight of Phenol = 50 g

~~#1 Beaker~~

#1 Beaker 30.0 − 28.2 = 1.8 at 100° C

#2 Beaker 28.8 under 1/2 gram 110–170

#3 Beaker 69.0 − 30.5 = 38.5 g. 180° C constant.

#4 Beaker 36.0 − 33.5 = 2.5 g 185–190° C

Residue Boiling above 190° C 57.0 − 55.5 = 1.5 grams

Exp. #559 100 cc of Phenol Drum ##
IV from Silver Lake. Distilled.

100° C	Total	350	18 cc Water + Phenol
	Glass #1	17.8	2 cc Settle on Bottom
		17.2	16 cc Water
100–170°C	Total	191	
	Glass #2	18.5	1 cc
		0.6	
178–180	Total	92.6	
	Glass #3	20.7	
		71.3	
183–190	Total	270	
at 183 Bumps Violently —	Glass #4	15.5	
		11.5 g	
	Total		
	Glass #5	18.5	

Total Retort 57.2 g
Residue 55.5
2.7 g Residue

Specific Gravity 107.5 at 25° C

Summary Water 100° C	17.2 g	= 18 cc − 2 cc = 16 cc
100–170	.6 g	= 1 cc
178–180	71.3 g	=
183–190	11.5 g	
Residue over 190	2.7	
	103.3	

107.5
103.3
4.2 Error.

The Boiling Point from Batch # 2 was 183–184 and did not crystallize. The Boiling Point of Drum # IV. is 178–180° C and crystallizes in the tube even when slightly warm. the fraction 183–190 Partly crystallizes and it looks as if 75% of that fraction were also good Phenol. The residue is also slight

Exp #560 a A Thimble 3/4 filled
Sand Sandstone
1 was extracted with Gasolene in the
Soxhlet Apparatus for 3 hours.
with Regular 62½° Bé. Gasolene.

The part insoluble in Gasolene is
560 a

560 b is the part soluble in gasolene
after distilling off the solvent.

560 c The Gasolene distilled off.

561 a Same as 560 a except used
Petroleum Benzene

561 b. Part sol in Benzene –

561 c The Benzene Distilled off.

Exp. #562 L.O.
Destillation of Vaselene given to me by Mr. Pinnickie.

46 1/2 is oil
51 1/2 g Vaselene 96 Vaselene (Solid)

at 70°C slight condensation nothing distilling over

at 90°C White fumes nothing going over.

At 105° C. Starts to distill but the thermometer gradually keeps rising without much coming over up to 200–220 then suddenly it goes up and keeps going up! Had to stop on account of danger breaking thermometer.

Sample #562 A from 105 to 220 Remains liquid smells like decomposed greases.

Sample #562 B what I could get above 220 up to limit of the

thermometer.

Exp. # 563 L.O.
Same as 562 except distilled in vaccuo = ~~104.5~~ 40. g.
Vacuum 27" heated up to 240°C = 463°F and nothing but a mere trace of anything distilled over this is 13°F above the maximum temperature to which the wax is heated.

Bottle #563 L.O. Vaselene which remained in the retort after bring up to 240°C –

562 Large Batch
Started at 180 then kept rising to 225 where it remained for a short time then gradually ~~was~~ keeps rising. Stopped at about 280°C when the drops on ~~the~~ a glass plate congealed.

#562 A Part which came over without congealing

#562 B Part remaining in retort.

Exp. #564

25 g Stearic Acid was distilled in a Vacuum of 27"
at 220 a little came over (trace)
while nearly all came over at 240°C
then the thermometer dropped to 220°C
and no more would come over
probably Retort to ~~Sur~~ Surge —

564 A. Part which came over at 240°C

564 B Part which remained in the Retort

Original Stearic Acid	M.P.	~~66°C~~ 55°C
#564 A.	" "	55°C
# 564 B.	" "	60°C

565 Sample of Na_2CO_3 and NaOH
NaOH Sp. G. @ 15°C = 1,2220
Alkalinity = 19.78%

14.7337
13.0860
1.6477 g Na_2CO_3

13.8440
13.0860
.7580 g NaOH

7.5
00
7.5

$$\frac{7.5 \times .020}{7580} = 19.78\% \text{ NaOH}$$

13.00
8.00
Phenolphthalein 5.00

17.80
8.00
9.8 M. Orange

$$\frac{9.8 \times .020}{1.6477} = 11.89\% \text{ as NaOH.}$$

X : 11.89 NaOH :: $Na_2CO_3 \cdot 10H_2O$ 286.3 : NaOH 80.2

$$\frac{11.89 \times 286.3}{80.2} = 42.55\% \ Na_2CO_3 \cdot 10H_2O$$

Sp. G. @ 15°C = 1,1707

$$\frac{.02 \times 5.0}{1.6477} = 6.0\%$$ combined with the CO_2 Radicle

565 Na_2CO_3 Sol.

~~This sol was made to correspond to the~~
This sol was made from C.P. Dry Carbonate of Soda and made to correspond in alkalinity by titration with H_2SO_4 to that made from the regular crystal soda from Hoffman.

15.6605	27.1	35.2
13.0860	20.0	20.0
2.5745 g	7.1 Phenolphthalein	15.2 Methyl orange

$$\frac{15.2 \times .02}{2.5745} = 11.80\% \text{ as } NaOH.$$

$$\frac{7.1 \times .02}{2.5745} = 5.51\%$$ combined NaOH with the CO_2 Radicle

Not used

Made larger sample of 500 cc
Titration

14.6620	71.35
13.0860	620
1.5760	9.35

$$\frac{9.35 \times 02}{15760} = 11.86\% \text{ NaOH}$$

566 Nov 7, 14

Stocking Sol. for Films.

#566 The regular #2 Solution Made for Mr. Grimes to stick Celluloid to Powder Blends

567 Celluloid Solution made by Blustein

acet anilid alcohol amyl acetate
568 $C_6H_5NHC_2H_3O$ + C_2H_5OH + CH_3CO_2
C^5H^{11} = Quick cement

569 Ethyl Salicylate 1 turn after 1 week

570 Glacial Acetic Acid. Quick Cement.

#571 Standard Oil Company's Petrolatum #.0 Very little comes over at low temperatures the thermometer takes a rapid and steady rise to 300 °C when it starts to distill. This part which distills over I stopped after about 20 cc came over as the drops became solid on a glass plate. after cooling the 20 cc became solid.

572 Standard Oil Company's Petrolatum #5 a little comes off at 100 steadily ~~gradually~~ and rapidly rises to 230 when it starts to distill stopped same as in #571 the 20 cc becoming solid on cooling.

Nov. 24. 14. Tested 1st sample #0 from 1 lb. can 2% comes over at low temperatures then the rest comes over at 290 °C.

573 C.P. Dry Sodium Carbonate from Bates Adamson. dissolved in water, saturated, at the boiling point then crystallized in the cold.

Then made up a solution to Standard by titration as in #565

* The sample of #0 from the large batch 5 lb can Nov 20, 14. distills the same as the sample but comes over about 8 to 10 degrees higher 278 – 300.

Slow or rapid distilling makes a difference as in the case of slow heating there for decomposition can take place more readily therefore both samples were distilled at about the same rate, as it is possible to do so.

Sample of Spindle oil Vacuum Oil Co Distills over at 310 °C. (Vapor) Thermometer in the oil 330 °C nothing below this temperature

Exp. # 574

Testing Aniline Dyes sol. in water to see if discolored by 30% H_2SO_4 — 1/2 Benzol Sulfonic Acid — 6 balls — and Plaster Paris

Cotton blue not discolored by H_2SO_4
but slightly by the benzol sulfonic acid
Totally by 6 balls
" discolored also by mixture from
filter press in the Carbolic Acid Mfg.

short = 6" strip with 1 joint
Long = 6 foot strip with 1 joint

#575 * Film Cement
20 g Acet Anilid in 100 cc Wood Alcohol (Absolute)

576 Same as #575 except added 4 cc Glacial Acetic Acid

577 1 g Film in 50 cc Amyl Acetate + 2 cc Glacial Acetic Acid.

578
~~579~~ 1 g Film dissolved in 100 cc Amyl Acetate + 5 cc Acetic Acid Glacial.

579 Film Dope Made by Blumenauer
25 cc + 1 cc Glacial Acetic Acid

580 Acet Anilid in Acetone 20%
20 g in 100 cc Sample to Halpin 2 short 1 Long 1 fair after wash

581 Same as 580 except + 10% Glacial Acetic

582 N cement from Celluloid Co
3 short cement to Halfin 1 long 1 hr. 39 min average

583 A.B. cement from Celluloid Co
3 short

584 1g Film in 50cc Amyl Acetate
+ 25cc Acetone + 2cc Acetic Acid
3 short. 1 fair after 1 week

585 75cc Acetone 25cc Amyl Acetate
20g Acetanilid. 1 long
3 short. 1 fair after 1 week.

586 Same as 585 except added
To 1 volume of 585 & 1 volume of Acetone
1 short

587 Same as # 580 except 1 vol of 580
and 1 vol Acetone 1 fair after 1 week
5 hrs 9 min average
20g Acetanilid in 200cc Acetone 10% Sol

588 1g Film in Acetone 25cc ✓

589 1g Film in Amyl Acetate 50cc ✓
1 strip cement to remedy 6 hrs 52 min
(distilled) 15 hrs 18 min longest

590 1g Film in Acetone 25cc + 1cc Acetic Acid ✓

591 1g Film + 25 Acetone + 25 Amyl Acetate ✓
1 strip cement to Remedy (7 air)

592 1g Film + 25 Acetone + 25 Amyl Acetate ✓
+ 1cc Glacial Acetic (Best.) all three OK
1 strip cement to Remedy after 1 week
2 hrs 49 min

593 Same as 588 except added g Camphor

594 Same as 589 except added g Camphor

2 hrs 37 min the equal parts
Amyl Acetate Acetone Amyl Acetate Good Method

595 Same as 590 except added g. Camphor

596 Same as 591 except added g Camphor

597 Same as 592 except added g Camphor

~~598 film 1g in 50cc Amyl Acetate 25cc + 1g Camphor.~~

598 Amyl Acetate 25cc + 1g Camphor

599 Acetone 25cc + 1g Camphor

600 Acetone 25 Amyl Acetate 25 + 2g Camphor
1 strip Cement to Kennedy

~~600~~ 601 Amyl Acetate the part which distills between 100 — 130 —
1 strip 5 samples.

602 Same as above only the second lot distilled. using 10cc of the distilled Amyl + 5cc acetone.

603 Same as 602 except substituted 1cc oleic acid in 25cc mix for the acetone

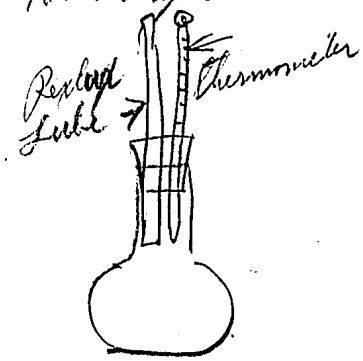
604 C.W. Cement Celluloid Co —
3 strips 1 loop sent to Kennedy

605 A.P. Cement Celluloid Co
3 strips 1 loop sent to Kennedy

606 Acetal

77

607 25 g S.G. Phenol
+ 13.5 g 40% Formaldehyde



( )

Put on bot flute 11.00 AM Saturday

Temp start. 30° C.

78

608 1g Film in Acetic Ether
1 hr 31 min

603 Experiment on Stearic Acid Impurities

65.0200 total
62.8145 glass
2.2055 g Stearic acid was dissolved in 200 cc alcohol then kept in the ice box over night then one hour in ice and salt then filtered off rapidly the first 50 cc of the filtrate because this was the coldest. of this 50 cc 25 cc was evaporated on the hot plate.

15.1520 total
15.0255 glass
.1265 g in 25 cc
8
1.0120 g in the 200 cc in total

A 2.2055 Total. or Original
B 1.0120 Soluble in alcohol at 0°-5°C
C 1.1935 Insoluble in the alcohol at 0°-5°C

Melting point of C.P. Stearic Acid = 69.2
" " " A = 59.0
" " " B = 53.0
" " " C = 64.0

1 gram of Stearic Acid required 7.5 cc $\frac{n}{2}$ NaOH

$\frac{1000 \times 2}{7.5} = 266.6$ mean combining weight.

Molecular weight of Stearic Acid = 284
" " " Oleic Acid = 256

$\frac{\text{Mean Combining Weight } 266.6}{284 - 256} = \frac{266.6}{.29}$

= 95.2% Stearic Acid

100.0
95.2 Stearic
4.8% Impurities as Palmitic

Molecular weight of Stearic Acid = 284
" " " Oleic " = $\frac{282}{2}$

610. 1 g Stearic acid titrated by Hübels method gave

~~Stearic 38.6 cc 1/10 N~~

~~Blank~~ -

Blank 38.6 cc 1/10 N Sulfite

Acid Stearic 36.7

1.9 cc difference

calculating that for each cc difference represents .9.

∴ As 282 parts oleic absorb 254 parts Iodine I₂ the Iodine absorption ÷ 0.9 gives the percentage of Oleic Acid

$$.9 \overline{)1.9} = 2.1\%$$

18
10
9
10

Precipitation method

3 g Stearic Acid with Lead Acetate 3 g in 100 cc Boiling water Stearic dissolved in Alcohol made neutral with alcoholic KOH then free washed hot water dried then let stand 10 hrs in ether filtered washed with ether. dried + weighed gave 4.000 g Pb Stearate

Pb Stearate Stearic Acid

386.5 : 284.4 : 4.00 : X

$$\frac{4 \times 284.4}{386.5} = 2.932$$

30000 grams used
2.9320

$$3.\overline{)06.80}$$ grams in filtrate

$$\frac{.0680}{3.} = \frac{06.80}{3.} = 2.2\%$$ Oleic acid

2nd coat at 4.30

Dec, 12, 14

# 611 Repetition of Copper coat
# 112 " Making of Fire Proof Paint
X = Marked by Mr. Edison as 2nd or Fair
Pieces of [illegible] 12" Long were painted on all sides with one or two coats of the following Solutions.
Then left dry ~~on Cot plate~~ for 12 hours about ~~[illegible]~~ in air then burnt on a wire ready for test.
All dried 1½ hrs except those of 2 coats (1st coat 2 hrs second coat 12 hrs.

611 One coat of Sodium Silicate of 1415 Sp. G. from Chas. Cooper. dried 12 hrs done

All samples dried as above unless otherwise Specified.

612 Same as 611 except 2 coats 2nd coat painted on 12 hrs after 1st coat. then dried 2nd coat 12 hrs ~~[illegible]~~ done

X 613 100g Na Silicate (Cooper) + 10g N.J. Zinc Co Special Zinc Oxide. One Coat dried 12 hrs Little thick to spread. done
Fair.

X 614 ~~50g Na Silicate (Cooper) +~~ Same as 613 except 2 coats dried same as with # 612 done
Best yet. (on one side) pretty good (on the other)

615 100g Na Silicate + 10g Chalk. 1 coat dried 12 hrs Spreads fair. covering qualities of chalk poor. done

616 Same as 615 except 2 coats dried as in # 612. done

617 Sol of Caseiu 30 g Caseiu in 200 cc Water ~~Sp. G.~~ + 10 cc 20% NaOH = a 1% 1 coat. Sol of NaOH + 30 g Caseiu. + 2 cc out of a 3% Sol Carbolic Acid to keep from becoming sour.

618 Same as 617 except 2 coats dried as in #612

619 Caseiu Sol. as in #617, 100 g except added 20 g N-J Zinc Co Special Zinc Oxide. 1 coat. Spreads easy cream gold (done)

620 Same as 619 except 2 coats dried as in 612 done

621 Caseiu Sol as in 617, 100 g + 20 g Chalk. 1 coat. done Covering quality of chalk poor. done

622 ~~Caseiu Sol as in 6~~ Same as 621 except two coats dried as in 612. 1 coat done

623 ✓ 50 g Na Silicate from Cooper 1415 Sp. G. + 50 cc Water Sp. G = 1200 1 coat Dried 12 hrs Too thin done

624 Same as 623 except 2 coats dried as in 612 done

625 ✓ 75 g Sodium Silicate 1415 Sp G
+ 25 cc Water Sp G. = 1300 1 coat
Spread good more body done

X 626 Same as 625 except 2 coats
dried as in #612. done
Just 7 air

627 1200 Sp. G. Na. Silicate 100 grams + 10 g
Zinc Oxide U-J-Zinc 60. 1 coat
Too thin (not enough zinc) done

628 Same as 627 except. 2 coats.
1 coat done

629. 1200 Sp. G. Na Silicate 100 grams + 20 g Zinc Oxide
1 coat. Thin not quite enough zinc
done

630 Same as 629 except 2 coats.
1 coat. done

631 1200 Sp. G. Na Silicate 100 g +
~~15~~ 30 g Zinc Oxide. 1 coat
good enough zinc done

632 Same as 631 except 2 coats.
1 coat done

633 1300 Sp. G. Na Silicate 100 g + 10 g Zinc Oxide 1 coat.
Thin. done

X 634 Same as 633 except 2 coats
done Fair

635 1300 Sp. G. Na Silicate 100 g + 20 g Zinc Oxide (1 coat).
Little thin not quite enough zinc.

636 Same as 635 except 2 coats
done

637 1300 Sp. G. Na Silicate 100 grams + 30 g Zinc Oxide. 1 coat good wing power spreads good.

638 Same as 637 except 2 coats.
done

639 100 Na Silicate (1415 Sp G. bottom) + 20 ZnO
1 coat too thick hard to spread.
done

X 640 Same as 639 Except 2 coats
done Fair

641 Na Silicate. (Cooper) + 20 g Chalk. 1 coat. Covering quality of chalk poor. done

X 642 Same as 641 Except 2 coats done Only just fair.

643 Pattons Fire Proof Paint. Patton Paint Company Newark N.J. 1 coat. Spreads Easily

X 644 Same as 643 except 2 coats Second coat absorbed nearly as quick as first coat done Hard to Spread 2nd coat. Scarcely Fair.

645 Jock Brothers #199 C.W. Paint Works Long Island City Office 320 5th Ave N.Y. 1 coat. ~~50~~ 90 g Powder + 40 cc Water no instructions dissolved Powder in a little water first breaking up lumps strained then diluted. Thru 50 mesh

646 Same as 645 except 2 coats. done 2nd coat has a tendency to dissolve off 1st coat.

647 Same as 645 except took the total amount 40 cc Water and added the ~~50~~ 90 g Powder to it, Strained 1 coat. Thru 50 mesh

648 Same as 647 Except 2 coats.
done

649 J. M. interior Fire Proof Paint.
Asbestos) ~~50~~ 100 g Dry Powder + 40 cc Water
mixed thus Dry Powder Poured on a little water
broke up lumps Strained then diluted. 1 coat
then 5 wash. done

500 more
had to add
not Early

650 Same as 649 except 2 coats.
done

651 J. M. John Manderville N.Y. 100
same as 649 except added ~~total of~~ ~~50~~ cc
~~water~~ grams Powder to the total of 40 cc
water 1 coat done

adjust 500 gr. more

652 Same as 651 except 2 coats.
done

653 J. M. Washable Sanitary weatherproof
Fire Proof Asbestos Paint. 50 g Powder
+ 40 cc water added a little water then
strained and diluted. 1 coat.

654 Same as 653 except 2 coats.
done

655 J. M. Paint same as #653 except.
added the 50 grams powder to the total
of 40 cc water strained 1 coat.

656 done Same as 655 except. 2 coats.

657 Duplicate of 611 Strength Silicate

658 Dup. of 612 Strength Silicate
Two coats

659 Dup. of 613 Silicate + 10 ZnO
Fired 25 sec

660 Dup of 614 Silicate + 10 ZnO
Two coats
Fired in 25 sec.

661 Dup. of 615 Silicate + 10 Chalk

662 Dup of 616 Silicate + 10 chalk
Two coats

663 Dup of 619 Casein + 20 zinc O

664 Dup. of 620 Casein + 20 ZnO.
two coats

665 Dup of 621 Casein + 20 Chalk

√ 666 Disp. of 622 brown + 20g blacks.
Two coats

667 Disp. of 623 1200 Sp. G. Silicate

√ 668 Disp of 624 1200 Sp. G. Silicate
2 coats

669 Disp of 625 1300 Sp G. Silicate

√ 670 Disp of 626 1300 Sp. G. Silicate
2 coats
Fired in 12 sec

671 Disp of 627 1200 Silicate + 10 gr O

√ 672 Disp of 628 1200 Silicate + 10 gr O
2 coats

673 Disp of 629 1200 Silicate + 20 gr O

√ 674 Disp of 630 1200 Silicate + 20 gr O
2 coats.

675 Disp. 631 1200 Silicate + 30 gr O

✓ 676 Dup of 632 1200 Silicate + 30 zn o.
2 coats.

677 Dup of 633 1300 Silicate + 10 zn o

✓ 678 Dup of 634 1300 Silicate + 10 zn o
2 coats Fired in 18 sec

679 Dup of 635 1300 Silicate + 20 zn o

✓ 680 Dup of 636 1300 Silicate + 20 zn o
2 coats

Continued Page 105

July 28, 14.

Analysis of Oil Recovered from Alli-Mineral in Cattle American Oil & Supply Co marked mineral oil.

Colour = Nearly water white
Sp. G. @ 70°F = 0.8568

Viscosity	1st Time	= 2 min 21 sec	201
	2nd " "	= 2 " 22 sec	202
	3rd " "	= 2 " 20	200
Average Viscosity @ 70°F.		= 2 Min 21 sec.	201 sec.

Viscosity	1st Time	=	$24\frac{2}{5}$ sec
"	2nd " "	=	25 sec
	3rd " "	=	$24\frac{1}{5}$ sec
Average Viscosity @ 210°F.		=	25 sec

Flash Point = 395

Saponification Value = None

Total = 47.1887
Glass = 45.5526
Oil = 1.6361

cc 76.2 Blank 41.5
cc 35
41.2 cc $\frac{N}{2}$ Alc. KOH.

41.5
41.2
Difference .3

Iodine Figure

Total 84.5880 Blank 39.5 cc Blank 34.3

Glass 82.8793 Test. 29.4

Gal 1.7087 Test. 27.5 4.9

12.0

Gal 60 5625 Iter = .01248 I.

Gal 59.7135

.5490 .01248 X 4.9 = X100

.6490

= 9.4 second time 1st time 8.7. Iodine figure

Acidity = Trace

Alkalinity = None

Viscosity after heating 10 hours at 212°F while a good stream of air was passed through

1st _____

2nd _____

3rd _____

Average Viscosity @ 70°F after treatment =

Para phenylendiamin.

1st making of p. nitro aniline -

Paraphenylendiamin

2nd 25 grams p nitro aniline were put into a flask with 400 cc Water thereto was added 150 cc, 50 cc at a time 22° Be Commercial Hydrochloric Acid. Filtered then 80 grams granulated Tin added while heating cool in a bowl of water about 10 grams at a time, let stand hours. Then diluted with 200 cc Water H_2S passed until all the tin was precipitated Filtered evaporated to cc ~~then let the Hydrochloric acid salt crystal out~~ neutralized with soda and distilled.

681 Dup of 637 1300 Silicate + 30 ZnO

682 Dup of 638 1300 Silicate + 30 ZnO
2 coats

683 Dup of 639 1415 Silicate + 20 ZnO

684 Dup of 640 1415 Silicate 20 ZnO
2 coats Fired in 20 sec.

685 Dup of 641 1415 Silicate + 20 g Chalk

686 Dup of 642 1415 Silicate + 20 g
Chalk. 2 coats Fired in 18 sec

687 Dup of 643 Pattons Paint.

688 Dup of 644 Pattons Paint
2 coats Fired in 16 sec.

689 Dup 645 ~~Pattons Paint~~
Lock Bros 199 C.W.

690 Dup 646 ~~Pattons Paint~~
Lock Bros 199 C.W.
2 coats

691 Duf of 647 Lock Bros 199 C.W.

692 Duf of 648 Lock Bros 199 C.W.
2 coats

693 Duf of 649 J. M Interior

694 Duf of 650 J. M Interior
2 coats

695 Duf 651 J M Interior

696 Duf 652 J M. Interior
2 coats

697 Duf 653 J M. Washable Sanitary

698 Duf of 654 J. M. Washable
Sanitary 2 coats

699 Duf of 655 J M. Washable Sanitary

700 Duf of 656 J. M. Washable Sanitary
2 coats

701 Flat White Sample from
Herman Fougner C.E. 110 W 40th St
see Letter Dated Dec 15, 14. 1 coat.
144 hours dry

702 Dup of 701

703 Dup of 701. Except 2 coats
72 hrs dry then second coat.

704 Dup of 703

Out 705 100 g 1415 Sp. G. [illegible] at 10 [illegible]
Calcium [illegible]. 1 coat.

Out 706 Same as 105 [illegible] 2 coats.

Out 707 75 g 1415 Sp. G. [illegible] 7 oz
Lead [illegible].
Out. Great Precip.

Out 708 Same as 701 [illegible]. 2 coats.
Out. Great Precip.

709 100 g 1415 Sp. G. Na Silicate +
10 g Calc. Acetate
~~Calcia Acetate + Na~~ Calc. Acetate +
Na Silicate = Na acetate + Calc Silicate
white precip. ~~Necessary~~ Mag. Some
Bessin was sheeted out and this
series

700 Borate Calc Mag. Bessin. give
fine white with Na Silicate forming
other and Brownishness = Calc. Borate
+ Na Silicate = Calcium Silicate + Na Borate.
: Whether this series was not met.

711 Wisson 5 g Absorbent cotton soaked in an
excess of 15% Na OH 3/4 hour then pressed
out until weight was 15 g then ~~hour~~
added 40 cc CS_2 and shook 3 hours. Let
stand one night decanted off excess CS_2
and added 100 cc H_2O Strained thru
40 mesh sieve.
~~Took of 111~~ Took 100 g 1415 Sp. G. Na
Silicate + 10 g ZnO + 30 g 711 Wisson
2 coats Pice about 3/8" from top
Wisson nearly all separates out shown to
Mr Edison He says try it anyhow.
Full of lumps very small

Very Light Red. Burned in 30 sec.

~~710~~ Sources 110 g [illegible]
2 coats Pice. [illegible] 3/16 [illegible] Sp
on metal drawer (contracts very much)
Fired 23 sec.

713 [illegible] 110 g [illegible] 80 g [illegible]
2 coats Pice. dense.

714 100 g 1415 Sp. G. Na Silicate
+ 10 g ZnO + 30 g of 2 sol. of
10 g Tragacanth in 300 cc H_2O. (heated)
2 coats.

115 Sources 114 [illegible]
Tragacanth Sol.

716 Same as 714 except 10g Isinglass Sol. 2 coats. too thin

X out 717 100g Na Silicate 1415 Sp. G. + 10g ZnO + 30g of a Sol of 20g Glue in 50cc H_2O (heated to boiling) 2 coats. a little glue goes in and it separates out By making the sol up thin more will go in this is detrimental to the waterproofing qualities of the Na Silicate. (cut out)

X out 718 Same as 717 only 20g of the Glue Sol. 2 coats. Adding NaOH does not help.

X out 719 Same as 717 only 10g of the Glue Sol. 2 coats.

720 100g 1415 Sp. G. Na Silicate + 10g ZnO + 20g of a Sol. of 20g Dextrine in 50cc H_2O. 2 coats.

721 Same as 720 except 20g of the Dextrine Sol. 2 coats.

722 Same as 720 except 10g of the Dextrine Sol. 2 coats.

723 Dup of 711

724 Dup of 712

725 Dup of 713

726 Dup. 714

727 Dup. 715

728 Dup 116

729 Dup. 720

730 Dup. 721

731 Dup. 722

732 John Mandeville [illegible] Finish Paint 2 coats Mixed with the [illegible] as top but [illegible] the [illegible] is [illegible] bills.

733 Same as 132.

734 50 g Chinese Wood Oil + 10 g Finely Powdered Alum. 1st coat 36 hrs
Dup. 735 12,21,14 6:00 PM Total 72 hrs

~~735~~ 736 Same as 734 except. 80 g Finely Powdered Alum. 1st coat 36 hrs
Dup. 737 12,21,14 6:00 PM Total 72 hrs

Part of 738 Dec. 21, 14.

~~[illegible]~~ Experiment on ~~H~~ Solvents for Tetra Chlor Naphthalene.

1 = dissolved 1 st.

Solvent	Rate	Remarks
Benzol	1	Easily Soluble
CCl_4	2	Easily Sol.
Acetone	trace	Slow (very)
Gasolene	dissol.	Slow (very)
Alcohol Denat.	Dissol.	Dissol.
Turpentine	3	Somewhat slow
Linseed Oil Boiled		Dissolves when heated on hot Plate
Linseed Oil Raw		" " " " " "
Linseed Oil Boiled		Cold.

738 ~~737~~ Both dissolved in hot Tetra Chlor Naphthalene for 10 minutes then crystallized.
Dup. 739 no dup made

740 ~~739~~ Stock solution of 150 grams Boiled Linseed Oil + 30 g Tetra Chlor Naphthalene + 15 cc Carbon Tetra Chloride 1st not 36 hrs
Dup. 741 12, 21, 600 P.M. Total 72 hrs

743 ~~734~~ 50 g Stock Sol. # 742 ~~732~~ + 10 g Finely Powdered Magnesium Sulfate
Dup. 743 12, 21, 600 P.M. 1st not 36 hrs
Total 72 hrs

744 ~~740~~ 50 g Stock Sol. # 740 ~~738~~ + 10 g Finely Powdered Sodium Silicate
~~Dup. 745~~ Dup not by mistake 1st not 36 hrs
12, 21, 600 P.M. Total 72 hrs
744 A Dup of 744 1st not 36 hrs

~~746 50 g. Acetic Acid + 20 grams Barbers Extra color naphthol + Extra chloride of barium~~

745. 40 grams of 740 + 20 grams Zn O. 1st coat 36 hrs.
746 Dup of 745 12, 22, 8.00 A.M.
2d c 72 hrs

747 H.W. Johns. Asbestos Waterproof Fireproof Paint. H.W. Johns Paint Mfg Co Newark N.J. 1st coat on 12, 22, 14, 1037 AM Dries rather quick. on 12 hrs.

748 Dup. of 747

⊕ no good

The following samples were tested by placing them in a hot muffel at a cherry red heat. and noting the time it took for them to catch fire

711	30 sec	⊕	702	7	sec
659	25 "	⊕	732	12	"
660	25 "	⊕	700	5	"
670	12 "		698	6	"
678	18 "		696	14	"
684	20 "		694	15	"
686	18 "		692	13	"
688	16 "		690	13	"
Pattons	17 "				
712	23 "	⊕			
714	22 "	⊕			
713	18 "				
722	20 "				
721	18 "				
720	13 "				
716	18 "				
715	15 "				

Exp. # 749 500 cc. of Automobile Gasolene from the Std. Oil Co.
distilled with use of the long fractionating column – Distillation Starts at 30°C

From	30 – 78°C	c.c.
From	78 – 80° C	c.c.
From	80 – 84° C	6 c.c.
From	84 –	

Mr. Edison says to stop as there was not enough of the benzole fraction and that which I got did not crystallize on cooling.

Exp. #

Exp # 750 500 cc Pennsylvania Crude ~~using~~ distilled using 21 inc atmosphere column for fractional Destillation

Between	40°C and 78°C	22 cc
"	78°C and 84°C	5 cc
"	84 and 90°C	3 cc
Total		30 cc

Distilled 3 more cuts ~~and~~ collecting the [illegible] 40°C and 90°C [illegible] together with the 3 distillate of the first batch.
Total = 130 cc between 40°C and 90°C from a total of 2000 cc. Crude oil.

Result.

40 to 78°C	66	Cubic Centimeters
78 to 84°C	22	" "
84 to 90° fraction which was left in still		40 Cubic Centimeters

Finally in still became to small —

751 Pattern #1 Fireproof Paint Red Dec 30/14
Sp. G. 1675 Olittle hard to spread 2 coats

752 Dup 751 2 coats

753 Pattern #2 Fireproof Paint Red Dec 30/14
Spreads like thick paint not as thick as
#1 2 coats

754 Dup 753 2 coats

#755 Dup. of 722 Dec 23, 14
Large Batch.

14,400 g Sp. Silicate
1440 g Zinc Oxide
1440 g Pulverized [illegible] =
540 g [illegible] in 900 cc H2O

755A

14,400 g [illegible]
1440 [illegible]
540 [illegible]

Pulverized [illegible]
[illegible] Zinc Oxide

32 lbs Na Silicate
3.2 lbs Zinc Oxide
1 lb 3¼ oz Pulverine

#756 Jan. 1, 1913.

Distillation of [illegible] of Oil from.

[illegible]

Distillations were made [illegible] fractionating [illegible].

From 200 cc of Oil

Distillation at 69°C

From 69 to 78°C 7 cc [illegible] oil

" 78 to 84°C 1 cc [illegible] oil

From 84 to 100°C [illegible]

6 lb [illegible]

[illegible]

Exp. #757

Received Jan 4, 15, from
U. S. Asphalt Refining Co N.Y. and
Baltimore (2) 5 gal samples Repeaus Co.

#757 19°Be Sample from the Petroleum de Ebano Wells 75 Miles south of Tampico 500 cc taken
Too small a quantity
Dest. commences at. 79 to 84 = 4 cc

Distilled 1000 cc in ordinary retort from 79 to 130°C

This was redistilled in the Fractionator Column 21 concentrations — and obtained
79 – 94 = 5 cc
94 – 108 = 10 cc
108 – 115 = 6 cc
Mostly all higher boiling side

Jan. 7, 15.

758 Distilled 1000 cc [illegible] side
about 2 cc came over between the ranges
79 to 130°C [illegible]
No [illegible]
No [illegible]

Jan 8, 15.

#759

Solubility of Beeswax and of Ceresin in various solvents. For test floating cement.
Order of solubility #1 being best

Solvent	Ceresin	Beeswax
Benzol.	#1 Trace sediment	#1 Clear
Benzine	5 3/4" Sediment	2 Clear
Toluene	4 1/2" Sediment	3/4" Sediment
Acetone	5 N.S.	N.S.
Turpentine	3 a little sediment	3 Clear
CCl_4	#2 a little sediment	2 Clear
Alcohol	N-S.	N-S.
	Acetone trace solid on cooling	Acetone and alcohol trace solid on cooling

Warmed in water bath to dissolve the Beeswax or Ceresin then let cool to room temp.

#760 Test of Wood Preservative Jan. 11, 15.

	Lights	Dark
Fires 150 Wards	5.9 g	1.3 g
Remaining on 1S7 Wards	6.2 g.	8.2 g
Fires 100 N.S.	5.2 g	7.7 g
Remaining on 100 Wards	1.5 g	5.3 g

#761 Saturation point of the following Jan 14, 15.
Ceresin in CCl_4, Turpentine
Beeswax in CCl_4, Turpentine.
10 cc of the sat. sol taken

Beeswax in CCl_4	Total	19.6	
	Dish	17.9	
	Dissolved	1.7	17.0%
Beeswax in Turpentine	Total	20.8	
	Dish	19.0	
	Dissolved	1.8	18%

Cresin in CCl_4 Total 20.5
Dish 19.5
Dissolved 1.0 = 10.0%

Cresin in Turpentine Total 20.0
Dish 18.3
Dissolved 1.7 = 17%

The waxes were dissolved ~~[illegible]~~ in warm solvent then cooled and filtered, 50 cc of the filtrate taken and evaporated the solvent weighed.

#762
Pyroxylin Sol.
20 g Tri Nitrated Cotton dissolved in 100 cc Alcohol & 100 cc Ether

#763 Pyroxylin Sol
6.5 grams dry Collodion Cotton in a mixture of 25 cc Ether + 25 cc Alcohol.

#764 Lima Oil as per our order #20844 received by Mr. E. M. Seybolt, Steel through Dr Mackenzie of the Texas Co

From 1000 cc Crude.
80-85 — 1/2 cc
107.115 5 cc
practically no benzol.

Jan 21, 15.

#765 Experiment on Dust Laying Compound for Cement Floors.

#765 10 g ZnO + 8 cc Syrupy Phosphoric Acid
Too little acid not enough to wet all the ZnO

766 10 g ZnO + 6 cc H_3PO_4 + 2 cc H_2O
Too little acid

767 10 g ZnO + 6 cc H_3PO_4 + 4 cc H_2O

768 10 g ZnO + 6 cc H_3PO_4 + 5 cc H_2O
Acid Liquid remains so for 10–15 Minutes then slowly sets.

769 10 g ZnO + 6 cc H_3PO_4 + 6 H_2O

All must be well mixed in a mortar otherwise it will lump. Bubbles very much when applied to the cement.
Cement sucks up all the liquid and then the mixture dries crumbly.

770 10 g ZnO + 5 cc $ZnCl_2$ + 14 H_2O
Dries Chalky.

771 10 g ZnO + 4 cc $ZnCl_2$ + 12 H_2O
Dries Chalky

772 10 g ZnO + 6 4 cc $ZnCl_2$ + 10 H_2O
Dries Chalky

773 10 g ZnO + 6 4 cc $ZnCl_2$ + 8 H_2O
Dries Fairly hard.

774 Sat. Sol of $ZnCl_2$ put on the cement then added ZnO by powdering over and spreading on with a spatula adding more $ZnCl_2$ Sol just to make it workable.

775 30% Sol of $MgCl_2$ = about 60% Sol $MgCl_2 + 6H_2O$.
mixed with flashly heated MgO to a thin paste Spread on the cement this is M.G. dries all checked.

776 Same as 774 except. 1V above Sat Sol of $ZnCl_2 + 3VH_2O$

778 Same as 774 except. 1V sat Sol $ZnCl_2 + 1V.H_2O$.

779 Same as 774 except. 3V Sat Sol $ZnCl_2 + 1VH_2O$.

Exp # 780 Filled 2 Square, round corner Tablet Jars 2 lb size, 3/4 Full of Phenol Resin Clear Sample from Mr. Hoffman broken in 1/2" size for Specimen Samples

Exp. 781 Filled 2 Tablet Jars as above with Phenol Redistilled from the Crystals from Silver Lake ~~Boiling Pe~~ Pickley Point = 179-180°C Melting point 41°C

Crystallized by pouring in Jars through a funnel ~~then~~ at about 50°-60°C let cool to about 45°C then dropped in a crystal of phenol.

Feb. 1, 15.
~~Jan 3~~

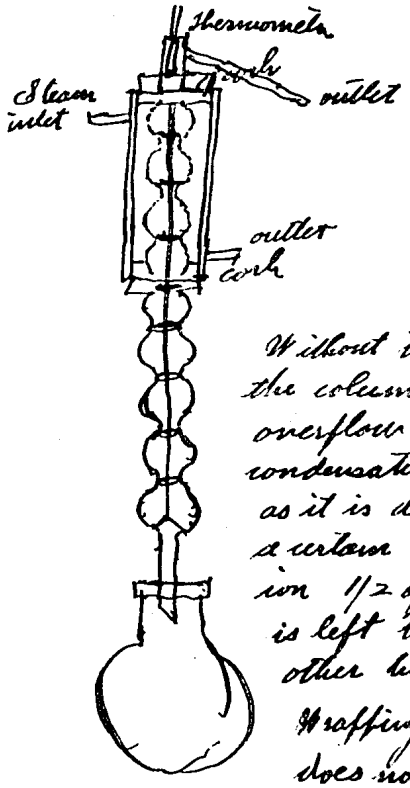
Exp. # 782
Distillation of ~~Phenol~~ Toluol from Silver Salse. 250 cc taken

Used a 500 cc capacity flask.

Rising thermometer to 110°C	=	2 cc
110°–112°	=	125
112°–115°	=	45
115°–120°	=	37
120°–125°	=	16
125°–130°	=	6
130°–137°	=	5
Not enough left to distill = Residue	=	9
Total		245 cc

Benzole distills at 80°C
Toluol " " 110°C
O Xylol " " 142°C
m " " " 137°C
P " " " 137°C

Exp. # 783 Same as # 782 except distilled toluol in still as per sketch



Without the steam jacket the column fills to the overflow – too much condensation.
as it is desirable to have a certain amount of condensation 1/2 of the condenser is left bare while the other half is steam jacketed
Wrapping asbestos around does not help.

column fills halfway therefore added gas engine cylinder oil and distilled some more then transferred to small still to finish the distillation

Results of #783

Column Still water jacketed

Rising thermometer to 110°C	= 11cc		11
Distilling at 110°C	= 134		
after adding heavy oil distills at 110°C	= 60		194
Transferred to small still	110–115 =	1cc	
Distilling at	115–120 =	2cc	
" "	126–130 =	8cc	
" "	130–137 =	8cc	
" "	137–150 =	16cc	35
Total			240

77% as Toluol — 250

240

Lost — 10cc

Second Result. 2000cc

Off to 119°.5	= 70 cc	
109.5 — 110.5	= 1630	80.0%
110.5 — 114	60	
Remaining above 114	= 220	
	1980	

loss 20cc

Sp. G. @ 15.5°C = .865

1st 5cc @ °C 95cc = °C

Fractionated the 1630cc.

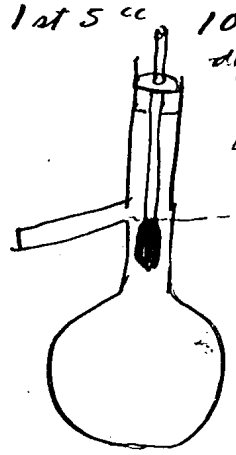
Below 110°C	195.	
110–110.8 —	457.cc	= 43%
Over 110°C —	977 cc.	

Sp. G. of the 110–110.8 = .866 @ 15.5°C

1st 5cc 109.9 — 95cc found over 111.5

difference = 1.6

Flask must be 200cc capacity with 100cc used for distillation



Level of top of Bulb

Sample distilled of Toluol which we had in the closet 1st 5cc at 110.8 at 95cc = 113.5 diff = 2.7

May 22/14

Exp. to Fireproof Fabric for Kinetophone Studio

#1 Dipped in Na. Silicate Sp. G. and wring out. Good

#2 Same as #1 except sponged after in dilute HCl then washed. N.G.

#3 1st sol Sat Sol Alum soaked, wring out, then put in 2nd Sol. of Ammonia. N.G.

#4 Same as #3 except soaked hot. Burns slowly

#5 Borax 12 grams, Epsom Salts 9 grams } H_2O 80 cc
Partially inflammable

#6 Ammonium Chloride 2 grams, Zinc Sulphate 1 gram } H_2O 20 cc
Good Not quite as good as #1

#7 Ammonium Phosphate 8 grams, " " Chloride 4 grams } 20% sol.
then in 20% sol Na Tungstate.
Good a little better than #1

#8 Alum 6 g, Borax 2 g, Na Tungstate 1 g, Na OH 1 g, Dextrine 1 g } 40 cc H_2O
N.G.

#9 Ammon Sulphate 8 g, Na Carbonate 2 1/2 g, Borax 1 1/4 g, Starch 2 g } 100 cc H_2O
Goods Boiled Hard Better than #1

#10 N.G.

#11 Magnesium Sulphate 10g H_2O 100g then in a sol of Ammonium Carbonate 10g + 5g Potassium + 100 H_2O
N.G.

#12 Same as #11 except Calcium Chloride for 1st sol.
N.G.

#13 Same as #11 except Zinc Sulphate for 1st sol.
N.G.

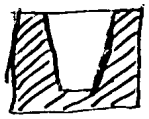
#14 Stannous Chloride 10g in 100 H_2O then in a sol of NH_4OH 20cc H_2O 100cc + 5g Potassium
N.G.

#15 Sol of Na Nitrate, N.G.

Exp. #784 Feb. 3, 15.

Melting of the fusion product from Silver Lake Phenol Plant. which consists of 100g Sulphonation Salt + 70g NaOH fused at 600 – 620°F. thick mush. If fused the fusion should seperate in two layers Na Phenolate on top and Na_2SO_4 + Na Sulphite on the bottom.

#784 Fusion made in a bath of lead in a heavy iron mould



#785 Same as 784 except added a little more NaOH no definite quantity

786 Made in a regular iron distilling pot.
85 g Fusion + 35 g NaOH Solid
Cooled slow Kept just fused for
15 minutes

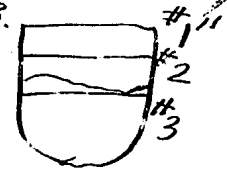
787 Same as # 786 except.
85 g Fusion + 50 g NaOH Solid
Cooled slow Kept just fused for
15 minutes.

788 Same as 786 except
85 g Fusion + 17 g NaOH.

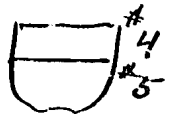
789 Same as 786 Except
Straight Fusion from Silver Lake

— # 786 Look double proportion
that is 170 g Fusion + 70 g NaOH solid
from Primary Battery Soda. come out
fairly easy from pot.
in two layers the
lower one brown the
upper one light.

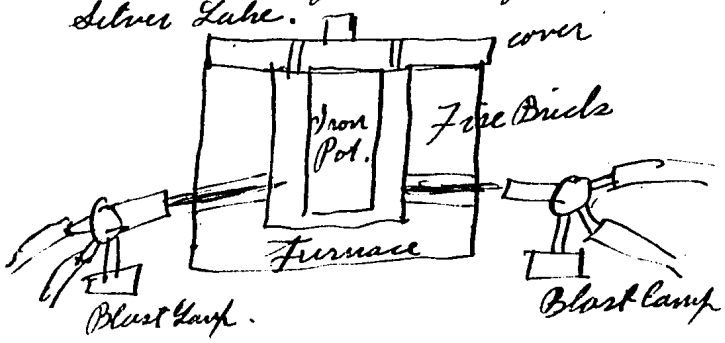
Mr. Edison divided into three layers
as follows #1 #2 #3.



787 Sawn in half
upper half #4 lower half #5



#788 Fusion of Fusion from Silver Lake.



Heated crucible in Furnace ~~then~~ a high heat ~~then~~ in order to get it hot throughout then let cool down to 600°F. Then put in 70 g Soda then gradually added the Fusion from Silver Lake 170 grams kept stirred then kept temp to 620°F. for 15 min shut off flame and let cool down.

Feb. 9, 15.

#789 Experiment to see if Viscose Cotton (cotton soaked in excess 15% NaOH then squeeze out excess of NaOH making cotton nearly dry) will absorb the CS_2 in Benzole making Viscose

One blank

#789 ~~100~~ 50cc Benzole + 2cc CS_2 + a good pinch of cotton shaken up and let stand over night.

151 Specification for Toluol
from British Government
Appearance to be clear water white liquid
free from suspended solid matter.
Sp. G. not to be less than .868 and not
more than .870 @ 15.5 °C
Boiling point must correspond approximately
to 110 °C corrected
Sulphuric Acid Test. 90 cc Toluol
shaken with 10 cc 90% H_2SO_4 should
impart only a slight colour to the acid
layer. after 5 minutes shaking.
Dist Test 100 cc are placed in a 200 cc
fractionating flask ~~of 200 cc~~
a thermometer divided in $\frac{1}{10}$° is so adjusted
that the top of the bulb is on a level
with side tube heat is applied over
a small area of the bottom and the
dist. conducted in such a way that
distinct drops pass over not a continuous
stream. After 5 cc pass over temp.
is read then read again when 95 cc
have passed over the variation must
not exceed 0.8 °C

790. Phenol was boiled with H_2O_2
using about 200 cc of 3% H_2O_2
for 25 g. Phenol in portions of
50 cc at a time boiled until water
nearly all driven off then added a
fresh lot of 50 cc.

This gives a blackish brown fairly
hard mass which is soluble
in denatured alcohol.

Dissolved in alcohol filtered and
then evaporated alcohol
Mr. Edson says that it is no good.

791

Distilled Phenol Special
sample given to me by Mr. Edison
Quantity taken 100 cc

Crystallizes Three layers Water + Phenol ?

100 – 110 °C		13	25 cc
110 – 120 °C		2	
120 – 140 °C		3	
140 – 160 °C		3 cc	
Liquid 160 – 178 °C		4 cc	
178 – 182 °C	Phenol	45 cc.	
182 – 190 °C		8 cc	14 cc
190 – 194 °C		4 cc	
Residue over 194 °C		2 cc	

Total as Phenol 178°C to 182°C = 45 cc
Total not phenol of 178–182°C = 39 cc

182–190 Does not cryst. readily but by throwing in a minute crystal from 178–182 cryst immediately
Loss 16 cc

792.
Took 500 cc Toluol just as it comes from Silver Lake (as residue of the Benzole still)

Shaken with 50 cc 90% H_2SO_4 10 min.
H_2SO_4 turns black Toluole means a little yellow
Seperated the H_2SO_4 from the Toluol.
then.
2nd time shaken 10 min. 50 cc 90% H_2SO_4
H_2SO_4 Dark red.
3rd time shaken 10 min 50 cc 90% H_2SO_4
H_2SO_4 Medium red — Toluol slightly red

4th time added 50 cc 90% H_2SO_4 let stand 1 hour on account of distillation #791 then shook 10 min
H_2SO_4 same colour as in 3rd time but Toluol wine red.

Washed with 1000 cc H_2O in 200 cc portions shaking 10 min. then let settle
Total washings of 4 instalments (nearly neutral
1.3 g H_2SO_4 absorbed
25 cc took 75.3 – 74.5 = .8 cc $\frac{1}{1}$ NaOH
= 1.3 H_2SO_4 would require 1.3 g NaOH.
x 2 = 2.6 g NaOH

After treatment with H_2SO_4 then washing with water 460 cc Remained making a loss of 40 cc = 8%

This 460 cc was then distilled in the water jacketed column Still.

80 – 110 + H_2O		104 cc
Water		43
80 – 110		92 cc
110 – 110.8		152 cc
110.8 – 112		

Result.

Temp. °C	No of cc.	+ H_2O %	%
80 – 10.9	– 66 – 12 = 54	14.3	11.7
109 – 110	– 38		8.2
110 – 110.8	– 152		33.
110.8 – 112	– 132		28.7
112 – 113	18		3.9
113 – 114	12		2.6
Over – 114	48		10.4
	466		98.5
		+ H_2O =	2.6
			101.1

33% or 152 cc corresponds to the Temp. required by the British Government while 61.7% can be reconed as good Toluol between 110°C. – 112°C. or for Hetter & Clerts from 109 – 112°C = 69.9% this corresponds to within .1% of their calculation

Exp. #793 100 cc Toluol from Silver Lake shaken with 2 cc 90% H_2SO_4 10 min let stand seperated H_2SO_4 put in new lot of 2 cc repeated operations.

After 5 lots the 4th and 5th lots of H_2SO_4 are ~~about~~ of the same shade of color the first lot being black the 4th & 5th light red –

This shows that the Toluol can be treated with 4 X 2 = 8 cc 90% H_2SO_4 for every 100 cc Toluol or 8%

Sp. G. 110. – 110.8 = 0.868 @ 15.5°C
" " 110.8 – 112 = 0.8685 @ 15.5°C

Distillation according to Specification of the Fraction 110°–110.8°.

after 1st 5 cc. passed over	109.8 °C	111.2
after 95 cc passed over	111.2° C	109.8
Variation	=	1.4
allowance	=	.8
Variation over the allowance		.6

The Fractions from

80 – 109	=	54 cc
109 – 110	=	38 cc
110 – 110.8	=	152 cc
110.8 – 112.	=	132 cc
Total	—	376
		362
loss		– 14

Total 362 cc were redistilled with corrected thermometer

From 80 – 110	= 120 cc	
110. – 110.8	= 194 cc	38.8
over 110.8	= 48 cc	

500 cc Toluol were treated with 4 lots of 10 cc each 95% H_2SO_4 Time 1/2 hour for each lot. Each lot the H_2SO_4 drained off in seperatory funnel. Then washed with 2 lots of 500 cc H_2O.

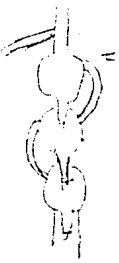
Total 465 cc

Then distilled in column still up to 113°C

	380 cc	Redistilled
1	up to 109	20
2	109 – 110	28
3	110 – 110.8	232 ✓ corrected.
4	over 110 –	32
5	Left.	62
		374

3rd distillation using the 110 – 110.8 Fraction 232 cc in a short condenser

110.1 – 110.9 = 110 – 110.8 Corrected	182 cc	= 36.4%
Below 110.1	= 34	
Over 110.9 left	= 14	
	230	



The 194cc Fraction Page 157 110. 110.8
Lost according to Governed test

Dist commences 109.5
5cc passed over 110
80cc " " 110.8 } rapid rise in these
95cc " " 112.5 } last 15cc

This means that 75% of the 194cc
leaves governed test

4)194 (48.5 = 145cc
16 3
34 145.5
32
20

500) 14500 (29%
1000
4500
4500

The 194 Fraction redistilled in small
condenser lost 18.4cc in flask.

Analysis of X

Part 1

Sulphates — a little aluminum

Silicates — none

Chlorides — all ?

Sp. G. 1250

Per Sulphates 18.00

Per Silicates 2.00

Per other 16.00